

# DESIGN RULES FOR REGENERATIVE FILTRATION SYSTEMS

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Regenerative adsorption systems can provide the user with the required level of chemical vapor protection continuously, for an indefinite period of time, without filter change-out. Two types of regenerative filtration systems, Thermal Swing Adsorption (TSA) and Pressure Swing Adsorption (PSA) have been studied for CW applications and evaluated at the prototype scale. Since these systems rely on both efficient adsorption AND efficient regeneration of the adsorbent, design requirements TSA and PSA can differ greatly from those used to design and operate current consumable filters. New design rules need to be developed to optimize and integrate regenerative air purification systems into host applications.

One critical aspect of regenerative adsorption systems is that optimum design is sometimes counter-intuitive. For example, unlike a consumable filter, optimum performance is not achieved by simply increasing the amount of adsorbent. Since equal consideration must be given to achieving rapid/efficient regeneration, the optimum design is typically achieved by selecting adsorbent(s) such that amount of chemical desorbed (per unit gas volume treated) divided by the amount of chemical adsorbed (per unit gas volume treated) is as large as possible. Examples of these effects are discussed as well as the implications for adsorbent selection.

## 1. INTRODUCTION

Proposed regenerable filtration systems for military air purification utilize adsorption to retain contaminant components. In order to provide continuous filtration duty, some fraction of the adsorbent inventory must be clean. These requirements can be met most readily using multiple adsorption beds. The details of the regeneration approach are used to characterize the various processing schemes. Typically some fraction of clean product air is directed to the regenerating bed in order to purge the offline adsorbent. When the driving force for regeneration is the pressure difference between the feed and purge steps, then the process is referred to as pressure swing adsorption (PSA) if the driving force is temperature difference, then the process is referred to as thermal swing adsorption (TSA). Many possible combinations and variants of this method can be considered.

While single-use adsorbent filters have been employed exclusively for chemical warfare protection, several concepts for regenerative adsorption filtration are now being considered. The principle design issues for selection of an advanced regenerative filtration method are the additional complexity and the added resource requirements. The availability of the required resource, e.g., pressurized air or heat source, is needed to make regenerable filtration a viable option for military applications.

## 2. PSA CASE STUDY

Although there have been several studies regarding the implementation of regenerative filtration for air purification, only one system has been developed beyond the prototype stage. The Comanche helicopter employs a PSA device to process air with the two-fold intention of providing a low humidity product appropriate for processing by an air cycle turbine. This system was originally configured as an

air drying system. Only within the last several years has it been evaluated for its chemical protection capability.

In most adsorption based filter designs, adsorbent selection is crucial in order to optimize the performance in terms of size and energy utilization. In air purification problems, this is largely due to the interference posed by adsorbed water. The requirement that the filter system provide protection against species with a wide range of physical properties also contributes to the need for thoughtful selection of adsorbents.

The following discussion describes the approach taken to optimize the Comanche PSA unit with respect to air drying. The most significant aspect of this approach is the methodology for adsorbent selection. The results demonstrate significance of proper adsorbent selection and its potential for system performance enhancement. PSA test conditions (see Table 1) were established based on system size and weight constraints, PSA feed conditions and the PSA effluent humidity requirement. This set of constraints and performance requirements represent a challenging problem since the water vapor separation ratio (feed water concentration divided by the effluent water concentration) is approximately 1000. Obvious difficulties limit the amount of experimental resources that can be devoted to testing a unit of this scale. Therefore, a series of laboratory scale studies were performed using scaled flow rates in order to investigate the type, ordering and quantity of adsorbent needed to meet the effluent water vapor requirement. This type of problem has been addressed previously in the development of adsorptive dryers. The design problem can be reduced to selecting those adsorbent(s) which will operate over a region in which the adsorption equilibria is favored for both adsorption and desorption. This is reflected by a linear isotherm shape. The adsorption equilibria for several common commercial adsorbents are presented in Figure 2. It can be seen that at low water partial pressure the 13X zeolite material has high capacity at a low water vapor pressure and therefore should be placed at the product end of the bed. Similarly the silica gel adsorbent exhibits a high loading change over most of the operating range of water partial pressures and therefore should be placed at feed end of the bed.

TABLE 1. PSA Conditions

Feed Flow Rate (Dry Air)	11.35 m <sup>3</sup> /min @ 273 K	440 SCFM @ 80 °F
Purge Flow Rate (Dry air)	3.69 m <sup>3</sup> /min @ 273 K	143 SCFM @ 80 °F
Product Flow Rate (Dry Air)	7.66 m <sup>3</sup> /min @ 273 K	297 SCFM @ 80 °F
Feed Temperature	330 K	134 °F
Purge Temperature	331 K	136 °F
Feed Pressure	5.37 x 10 <sup>5</sup> Pa	78 psia
Purge Pressure	1.16 x 10 <sup>5</sup> Pa	16.8 psia
Feed Water Partial Pressure	1.69 x 10 <sup>4</sup> Pa	2.46 psia
<b>Product Water Partial Pressure Target</b>	<b>13 Pa</b>	<b>230 K Frost Point</b>

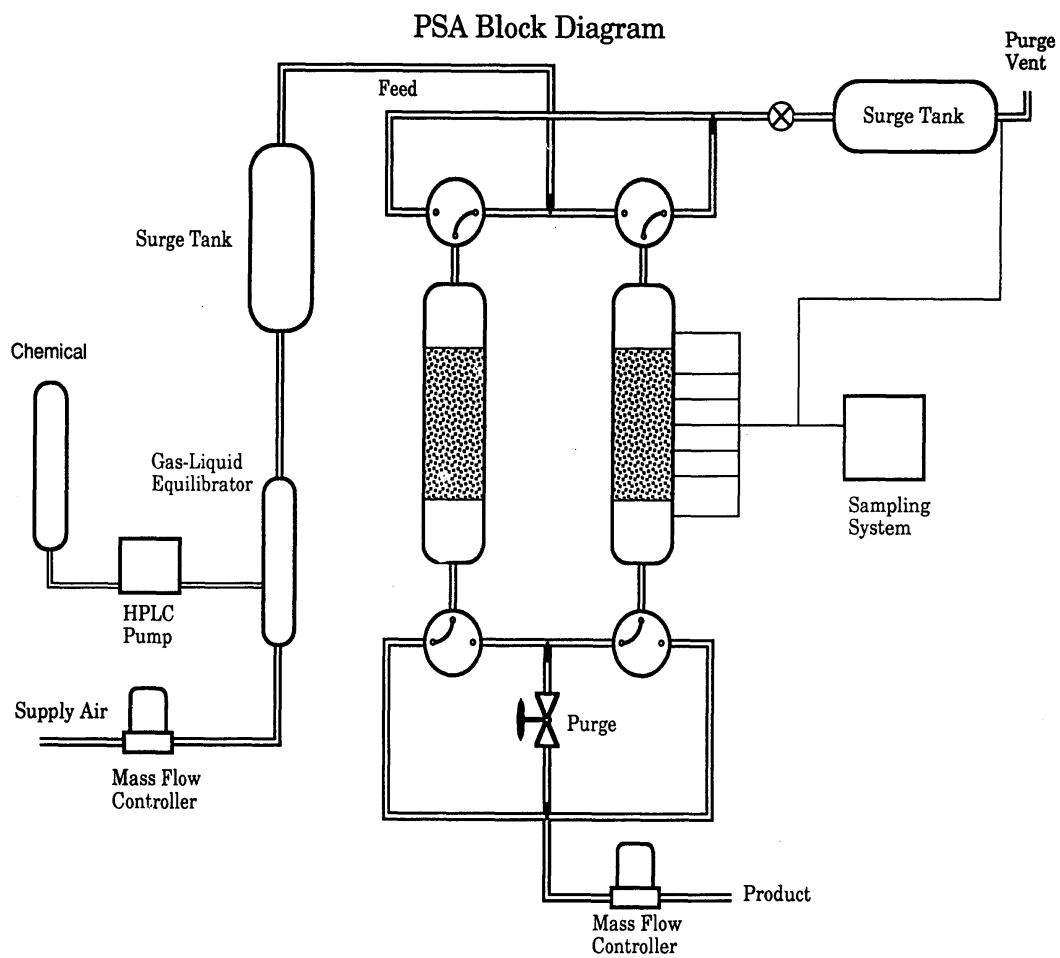


Figure 1. PSA schematic.

### Water Adsorption Isotherms for Silica Gel, Activated Alumina and 13X Molecular Sieve at 52°C

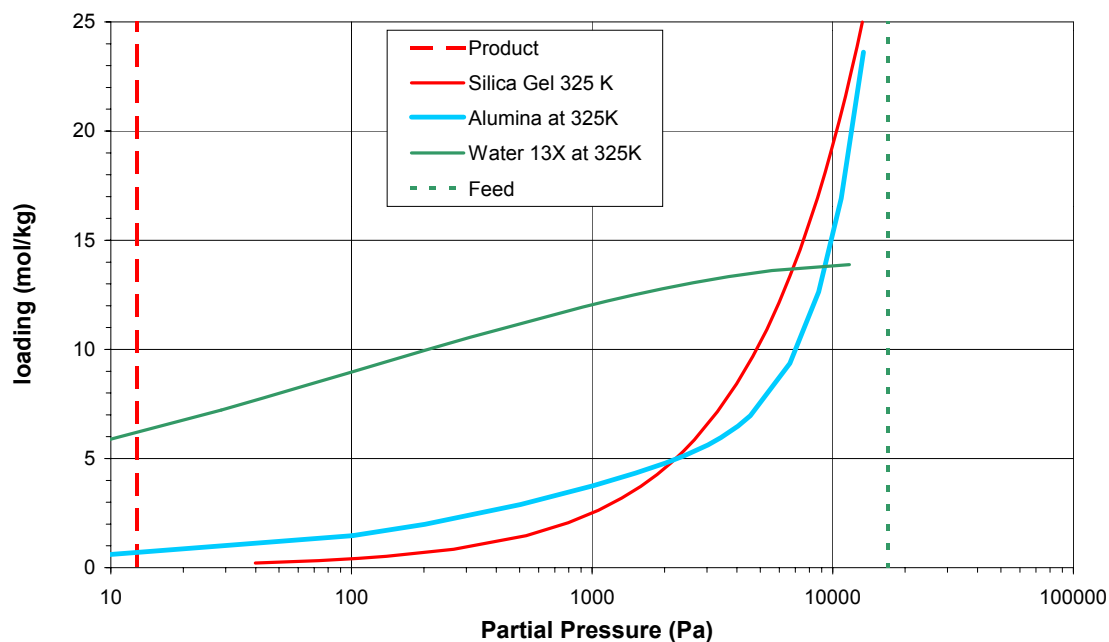


Figure 2. Water adsorption equilibria.

The results of several parametric tests on the lab-scale PSA unit are presented in Table 2. Decreasing product frost point is observed with the three-layered bed relative to all 13X. Also it can be seen in runs 2-6 that a higher silica gel distribution is favored and that a layer of small particle size silica provides increased separation due to better mass transfer rates. In each case considered, the total bed volume remained constant. Thus, by selecting the appropriate adsorbents and placing them in an optimum position within the bed, the product frost point was lowered from 263 K to 220 K, for a 43K improvement.

TABLE 2. Summary of Results for Layered Beds

Exp #	Product Frost Point T	Bed Configuration (from the Feed End)			
		Layer 1	Layer 2	Layer 3	Layer 4
1	263 K	All 10x20 13X			
2	248 K	35% 7x14 AA	35% 6x12 SG	30% 10x20 13X	
3	239 K	35% 7x14 AA	44% 6x12 SG	21% 10x20 13X	
4	239 K	21% 7x14 AA	62% 10x12 SG	17% 10x20 13X	
5	230 K	19% 7x14 AA	38% 10x12 SG	19% 18x25 SG	24% 16x20 13X
6*	220 K	19% 7x14 AA	38% 10x12 SG	19% 18x25 SG	24% 16x20 13X

### 3. TSA CASE STUDY

The optimum bed design and operating conditions for thermal swing adsorption differ from those of pressure swing adsorption. Although heating the adsorbent can allow the purge gas to be much more efficient, the thermal lag due to heat capacity effects and adsorbed water can significantly impact system performance.

A series of experiments were performed using the conditions listed in Table 3 with a laboratory-scale TSA apparatus. The cycle times for each test were selected using the following approach. First a column filled with the selected adsorbent was challenged until breakthrough to establish the maximum feed time allowed. Next, a column was filled with clean, dry adsorbent and allowed to come to equilibrium with the feed RH (no chemical). The column was then heated using clean, dry purge gas. The progress of the temperature wave through the bed was monitored using in-bed thermocouples. When the temperature change of the probe located 1.8 cm from the feed end (4.2 cm from the purge inlet end) reaches 2/3 of the temperature difference between the starting bed temperature and the purge inlet temperature, the heat is turned off and ambient purge gas is used to cool the bed. This established the heat time and cool time needed for each experiment.

The two cases, Exp. 4 and 2, represent dry and humid conditions using PICA carbon. This adsorbent exhibits high adsorption capacity for high volatility vapors due to a large microporosity, however it also exhibits a significant water adsorption capacity. It can be seen that the initial heat time for the humid case is three times that for the dry case. The cool time is only slightly longer than the humid case because the beds have been dried to a similar amount in both cases.

TABLE 3. Experimental TSA Conditions

	<b>Exp. #4-HCWC</b>	<b>Exp. #2-DCWC</b>
<b>Date</b>	<b>8/07/02</b>	<b>8/19/02</b>
<b>Adsorbent</b>	<b>Pica Carbon</b>	<b>Pica Carbon</b>
<b>Adsorbate</b>	<b>Chloroethane</b>	<b>Chloroethane</b>
<b>Feed Velocity (cm/sec)</b>	45	45
<b>Feed Flow Rate (SLPM)</b>	123	123
<b>Obsd Feed Temperature (°C)</b>	15.3	16.4
<b>Purge Velocity (cm/sec)</b>	15	15
<b>Purge Flow Rate (SLPM)</b>	41	41
<b>Purge Temperature (°C)</b>	80	80
<b>Feed Pressure (psig)</b>	4.12	4.18
<b>RH at Feed Pressure (%)</b>	61	<1
<b>Target Feed RH (%)</b>	60	
<b>Feed Concentration (mg/m<sup>3</sup>)</b>	2420	2399
<b>Feed Concentration Time (min)</b>	37	37
<b>Initial Heat Time (sec)</b>	340	120
<b>Cool Time (sec)</b>	642	420
<b>Cycle Time (sec)</b>	1964	1080

The temperature profiles recorded for the humid case is shown in Figure 3. The rapid temperature rise associated with the product end heater can be seen. Also note that the temperature recorded at the 1.8 cm probe reaches the purge temperature for only a few seconds. This highlights one difficulty with this method of purge heating. Whereas the most strongly adsorbed species are adsorbed at

the feed end of the bed, the product end of the bed is subjected to the highest temperatures. The effect of the thermal cycle on chemical filtration performance is presented in Figure 4. Good agreement is noted between the measured purge concentration using the continuous and discrete sampling. Only one discrete feed concentration sample was recorded but it does indicate that the feed concentration was  $2,000 \text{ mg/m}^3$  for the finite duration pulse of 37 min. The peak concentration in the purge stream is reduced by an order of magnitude after the second half cycle but still does not reduce another order of magnitude for the next ten half cycles. This is a result of not purging the feed end of the bed to the peak temperature. Also the presence of the feed chemical was observed in the product stream at  $5 \text{ mg/m}^3$ .

Temperature profiles for the dry case are presented in Figure 5 where all conditions are identical to those of the humid experiment except for the feed water concentration. The temperature wave moves much faster through the bed since there is not a requirement to supply energy for water desorption. The chemical performance is presented in Figure 6. In the dry experiment no breakthrough of chloroethane to the product is observed. The purge concentration profile also has a different behavior. Higher purge concentrations are noted for the dry case but these are of shorter duration resulting from the reduced purge half cycle and more efficient use of the purge gas. These two experiments demonstrate the effect that adsorbed water contributes to filtration performance. The appropriate selection of conditions and adsorbents can serve to minimize the impact of ambient humidity.

## CONCLUSIONS

The operating conditions appropriate for adsorption-based regenerative filtration have been demonstrated through an analysis of laboratory-scale studies. Adsorbent selection is critical to improved (optimized) regenerable system performance. For adsorption-based regenerable systems, adsorbed water can adversely affect system performance. More efficient heating and cooling methods can reduce purge flow requirements and/or regeneration time.

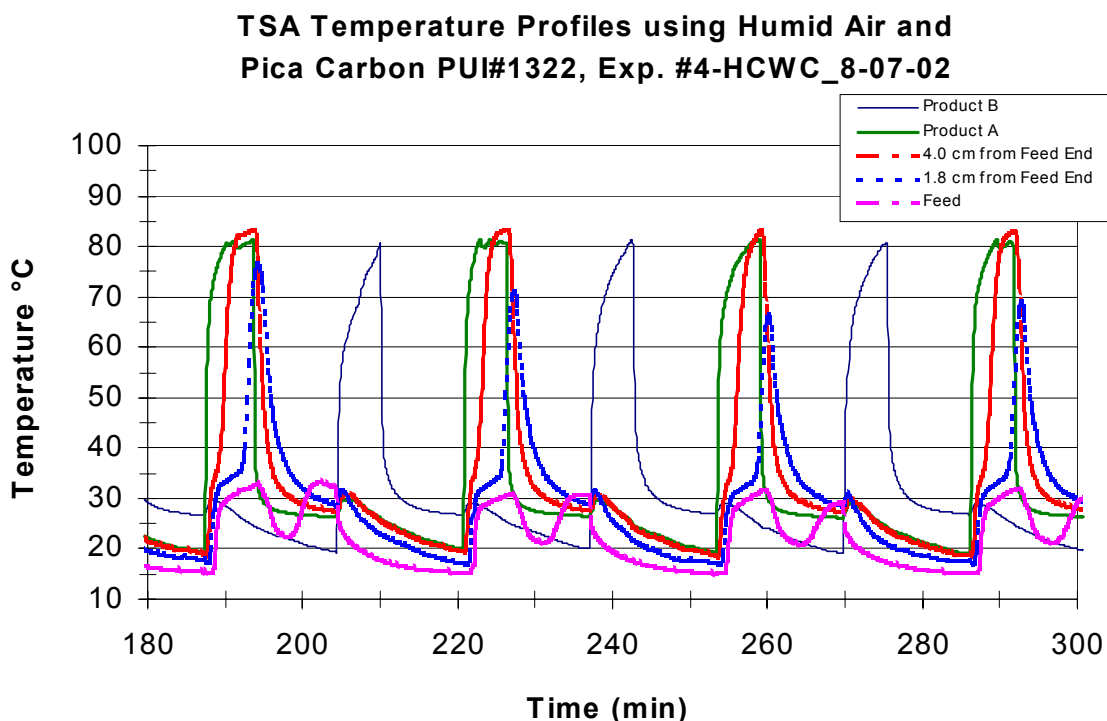


Figure 3. TSA temperature profiles for humid case.

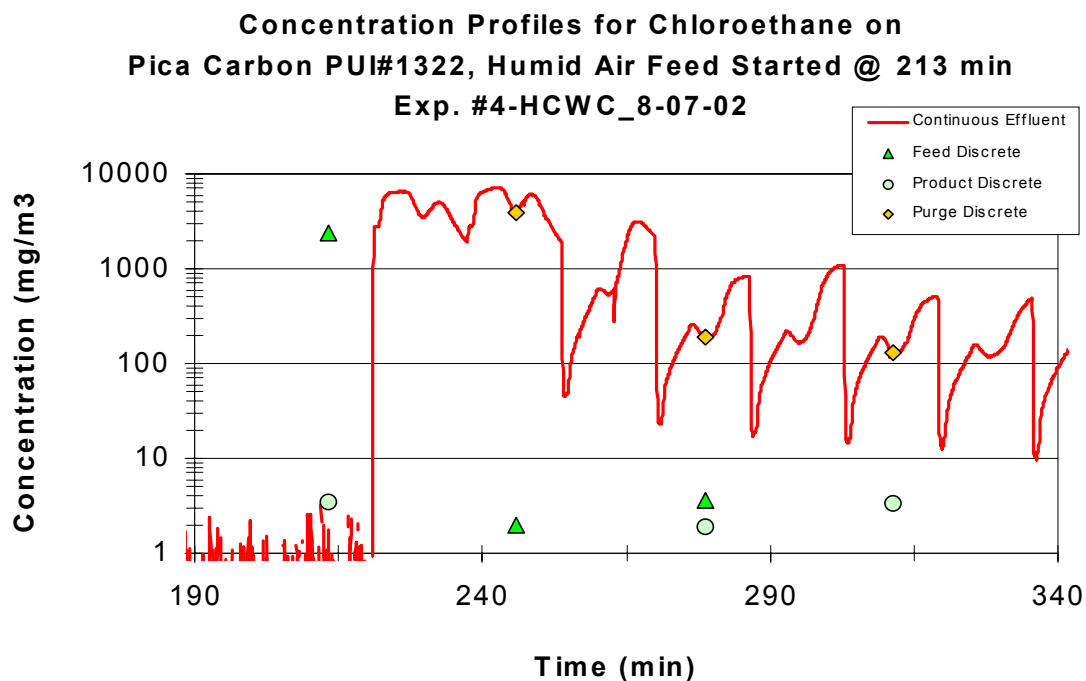


Figure 4. Temperature profiles for humid experiment.

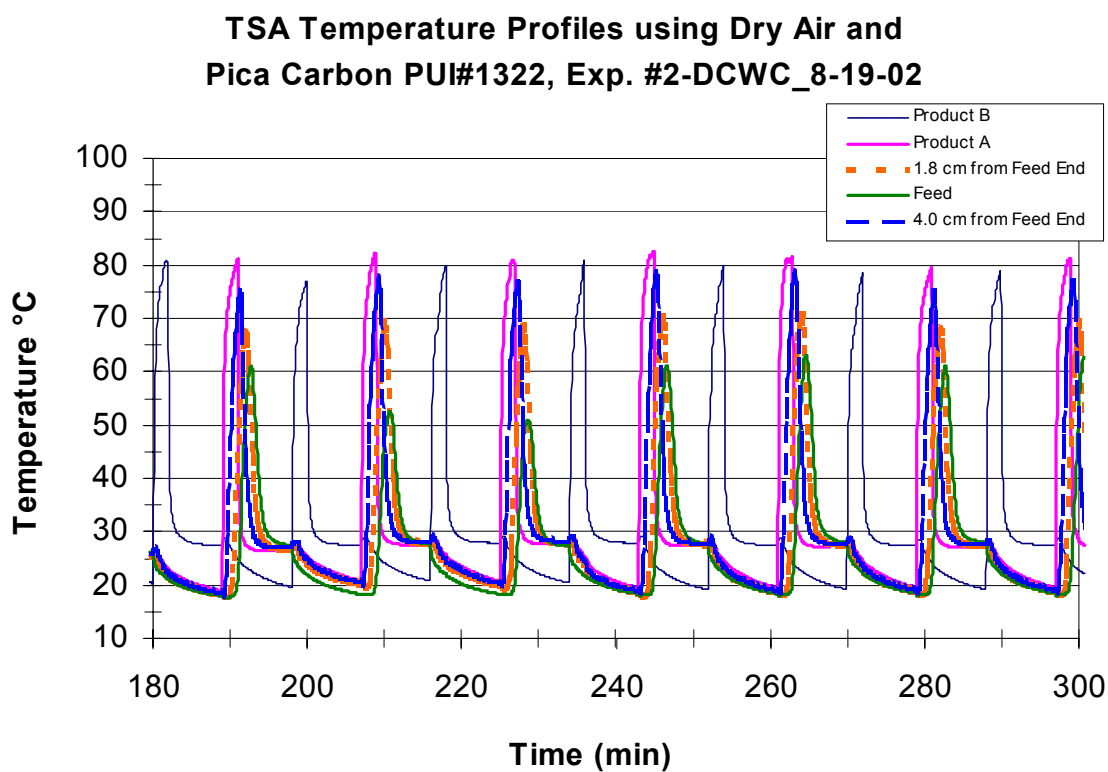


Figure 5. TSA temperature profiles for dry case.

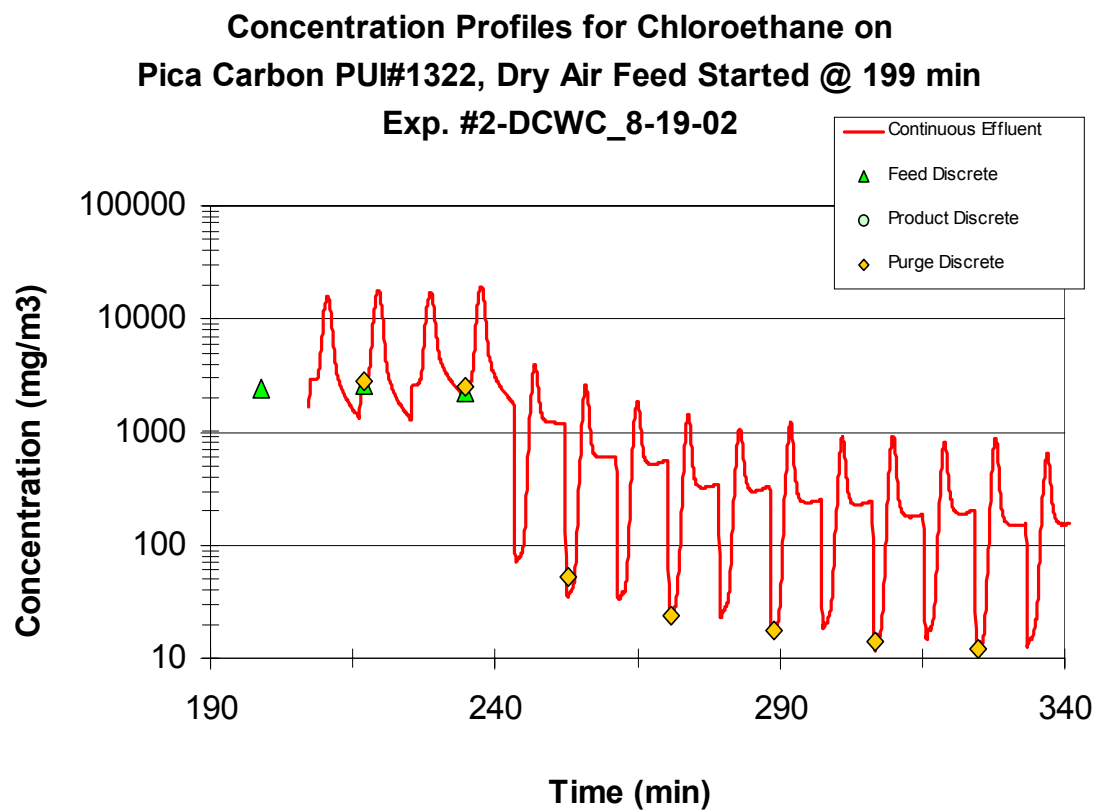


Figure 6. TSA concentration profiles for dry case.